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Transaltion of letters patent, FR 1,421,033**Improved ammonia generators, in particular for masers***(Invention: Morton Robinson)*

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*THE GOVERNMENT OF THE UNITED STATES OF AMERICA represented by the
Administration Nationale de l'Aéronautique et de l'Espace
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*(Application for patent filed in the United States of America on 24 January 1964 under No.
340,113 in the name of the present applicant.)*

The present invention relates to an apparatus as such for producing an ammonia gas and more specifically it concerns a source of such gas which presents itself in solid form and which is susceptible for use in ammonia beam masers and other similar devices.

Numerous tests and other technical operations aiming at space flights require extreme accuracy of measuring standards or other time-measuring means. More specifically, reference is made to research within the field of relativity phenomena which requires that time is determined with a precision within the range ensured by an ammonia beam maser.

It is known that a maser is a coherent amplification or production device for electromagnetic waves by excitation energy in atomic or molecular resonance systems. Such device uses an instable unit of atoms or molecules susceptible of being stimulated by an electromagnetic wave to radiate its excess energy at the same frequency and in the same phase as the stimulating wave, thereby ensuring a coherent amplification of same. Molecular radiation masers are well known in the art. A description of their principles of operation will appear from articles published in "Physical

Review", vol. 39, pages 1264 through 1274 (15 August 1955) and in "Proceedings of I.R.E.", Vol. 45, pages 291 through 316 (March 1957).

5 The first of such devices to be realized was the gas maser. Its functioning requires that it is supplied at a known and adjustable gas rate. So far, in the context of ammonia masers, a liquefied gas reserve has been used to that effect, but due to the strong vapour pressure of that liquid (approx. 9 kg/cm² at ambient temperature), it was necessary to enclose it in heavy stainless steel cylinders provided with complex pressure regulating valves. Although
10 various mechanical embodiments are known that are able to fulfill the above function, they are all associated with the drawback of weight and risk of defective functioning. According to one exemplary embodiment, the ammonia gas coming from the pressurized reservoir through pressure regulating valves leaves by a small opening in a vacuum container thus constituting a
15 molecular beam. Those of the molecules that are at an inferior energy level are deviated from the axis of separation or concentration of the apparatus by non-homogeneous electric fields that act upon their dipolar moment. Conversely, the molecules that are present on the superior energy level are guided towards the axis and transmitted to a resonance cavity for
20 microwaves. If the losses through the walls of the cavity and through the coupling openings are poor, or if the number of molecules is sufficiently high, an amplification or oscillation occurs. The ammonia beam maser is particularly useful as frequency standard or time standard due to the pointed and constant nature of the beam resonances. It has been found to be very
25 useful as frequency standard in systems for guiding satellites and missiles, where weight constitutes a major concern when deciding which type of maser to use.

30 US patent No. 3,013,218 filed on 14 May 1959 and issued on 12 December 1959 and issued on 12 December 1961 also describes an ammonia beam maser. It is the object of the present invention to realize an original source of

the gas which presents itself in solid state and contributes to further improving the advantages of the maser as frequency standard in a satellite or elsewhere. According to the present invention the ammonia gas is stored in a state from which it can be released slowly at a controlled rate. To this effect
5 the gas is transformed into a complex compound by means of a salt, that is a silver chloride. Since the latter constitutes a pure substance with a controlled equilibrium temperature and pressure, the release of ammonia becomes a predictable and controllable phenomenon. This characteristic feature is precisely the one required by the maser in order to function optimally.

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As already mentioned above, in an ammonium beam maser of classic type the amount of that gas in the apparatus is regulated by an external source comprising in principle a pressurized bottle of liquefied ammonia with valves, manometers, pumps, fittings and other accessories known in the art. In a
15 control system for satellite or missile, where the engine may continuously flip or turn about itself, such ammonium source should be eliminated since it is not possible to physically separate the gas from the liquid and to ensure that constancy of the rate of the gas phase should at any time be in contact with the regulating mechanism of that rate. Conversely, the compound of
20 ammonia solid matter according to the invention obviates this difficulty since it is possible to separate it from its vapour quite simply by enclosing it in a screen with meshes that are smaller than the dimensions of the solid particle.

As it can be predicted accurately, the decomposition mechanism of the salt
25 complex, which is the mono-ammoniated silver chloride, it is possible to use the salt in question, in accordance with the invention, to avoid the drawbacks of the known devices. The technique that takes place therein is automated in the sense that it is based on a chemical reaction rate that remains fixed.

It is thus the object of the invention to provide an apparatus and means enabling storage of ammonia gas in a state in which it can readily be released at an adjustable rate.

5 The invention further relates to:

Allowing the provision of a method and means for transforming ammonia into a complex compound of silver chloride and for subsequently releasing it in gaseous state based on the latter;

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Providing a novel source of ammonia gas for masers or analogue devices;

Providing such source of ammonia to masers that hereby become lighter, safer and less complicated than the sources used so far;

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Providing a novel source of ammonia gas for masers or the like that are susceptible to function in any position and which can be used on a mobile engine;

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Providing a novel chemical solid source of ammonia gas by which the drawbacks of the methods and devices used so far for the same purpose are obviated.

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The accompanying drawing being exclusively exemplary serves to improve the understanding of the invention, its characteristics and the advantages provided thereby:

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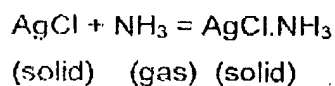
The only figure of the drawing shows in sectional view an apparatus for generating ammonia in accordance with the invention used for supply to a maser.

The complex mono-ammoniated silver chloride salt, designated by 1 in the drawing, is prepared in a copper sphere coated by glass. The sphere proper comprises the glass coating 2 and the copper plating 3. In the following, it is designated the reservoir. It may comprise an exterior insulating coating 10 of convenient nature as will be understood by every person skilled in the art. This sphere or reservoir has a mouthing 4 by which it is possible to fill it with salt and which also serves as exit for the ammonia gas. The mouthing 4 may comprise a screen 5 with meshes that are finer than the granulometric dimension of the salt 1 in order to prevent the particles thereof from being entrained in the connecting canalization system 6 and in other portions of the system. The reservoir is surrounded by an electric heater element 7 and a heat-detecting wire 8 of any expedient kind. The wire 8 constitutes one of the branches in a Wheatstone bridge. The circuit in this bridge requires, in a manner known per se, supply of energy to the heater element in order to permit it to uphold its constant temperature of about $+ 0,04^{\circ}\text{C}$. A connecting cable 9 connects the element 7 and the wire 8 to a an external thermostatic apparatus 11 that encloses the mentioned bridge and the electric source.

The canalization system 6 is connected to a tube 12 by means of a stop tap 13. The tube 12 is, in turn, connected, on the one hand, to a capillary derivation 14 and, on the other, to a second tap 15, the one or the other of which debouching in another tube 16 to enable the tap 15 to serve as bypass for the derivation 14. A sealing fitting 17 enables fixation of the above-mentioned apparatus to an accessory and to dismount it there from. During preparation of the salt 1, one may connect the apparatus to a source of ammonia gas, which bonds to the silver chloride to form the salt complex AgCl.NH_3 . It is well known in the art that a number of salts, when brought into contact with ammonia anhydride at low temperatures, cause the formation of compounds enclosing several ammonia molecules. When the complex salts thus obtained are heated, the ammonia thereof is released at a rate that depends on the temperature. In the exercise of the present invention the

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starting salt is silver chloride and to this ammonia molecules are added. The mono-ammoniated silver chloride ($\text{AgCl} \cdot \text{NH}_3$) thus obtained is in the state of a solid substance constituted by combination of the silver chloride anhydride (AgCl) with the ammonia gas anhydride (NH_3), in accordance with the formula:



10 When the silver chloride has thus been saturated with ammonia to almost 100 %, the tap 13 is closed in order to cut off the source from the atmosphere.

The capillary derivation 14 mounted on the apparatus is provided to ensure a rate of 10^{17} molecules per second at a predetermined pressure of ammonia. The apparatus thus prepared is fitted to the radiation chamber 18 of the maser by means of a sealing fitting 17 mounted on a canalization 19 provided therefor. Once this mounting is accomplished, the vacuum is established by keeping the tap 13 closed and the tap 15 open. The tap 15 which constitutes a bypass relative to the capillary derivation 14 is usually open during the charging of the source, but it should be closed before the apparatus is taken into operation. The reservoir is allowed to assume a predetermined temperature corresponding to the one calculated to ensure that the optimal power is accomplished; the tap 13 is opened and one may immediately obtain a maser signal there from. The gas travels into the chamber 18 by the canalization 19 and the tubing 20. The very strong resistance of the capillary passage 14 prevents any reasonable variations in the rate along the tubing 20 due to modifications in the ambient temperature. Thus an extremely stable ammonia beam is thus obtained which is upheld until all of the ammonia contained in the reservoir has been used completely.

One molecule (or mole) of AgCl.NH_3 , weighing 161 g, may release at a constant rate of 10^{17} molecules/second, which is a sufficient ammonia amount to run a maser for 70 days. Owing to its poor vapour pressure, the substance may be enclosed in a simple and light container, such as the
5 reservoir described above. Such poor pressure value, permitting to ensure temperature regulation as well as to determine the rate by means of a simple capillary tube, ensures that the apparatus has all the characteristic features required for an excellent solid source for ammonia masers.

10 In the foregoing description mono-ammoniated silver chloride is mentioned as the preferred constituent of ammonia salt complex. It is, of course, possible to employ other salts of the same kind. To illustrate by way of example, ammoniated acid salts in solid state are easily dissociated rendering, on the one hand, ammonia gas, and, on the other, free acid. If the
15 ammoniated solid complex is heated in a closed container, an ammonia pressure which is well determined in the presence of this salt will be established. That pressure will depend exclusively on the temperature and, if the acid or the metal salt is not volatile, it can be designated the dissociation pressure at the temperature concerned.

20 It will be understood from the foregoing description that the invention makes it possible to avoid massive sources of pressurized liquid ammonia for the operation of masers in satellites and other space engines. In the system according to the invention, the ammonia pressure is approximately equal to
25 1/10 of the atmospheric pressure at ambient temperature and it stays constant at the temperature concerned for as long as the two solid phases and gas are jointly present in the reservoir. Consequently the rate itself in the maser chamber remains constant until the entire amount of ammonia available has been used completely.

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Besides, it will be understood that the foregoing description was given exclusively by way of example and that it does not in any way limit the scope of the invention, which is not exceeded if its details of execution are replaced by any other equivalent. Also, obviously, the invention encompasses both the ammonia generators of the described type and the method of producing the gas by use of a solid salt by which they are executed.

RESUME

I. A method of supplying an appliance using gas and more specifically a maser with ammonia gas, consisting of charging a compound, such as an acid, an ammonium salt or another such metal salt and in particular silver chloride, with ammonia, to enclose the resulting ammoniated complex in a reservoir, and subsequently to heat the reservoir to determine the decomposition of the complex and the release of the ammonia absorbed therein, which depleted complex can be recharged in the reservoir as such following cooling thereof.

II. An apparatus for generating ammonia gas, in particular for masers, comprising essentially a reservoir containing an ammonium salt or a complex salt of a metal as such and ammonia in solid state, and means for maintaining the reservoir at a determined temperature at which the compound contained in the reservoir decomposes while liberating ammonia gas under a pressure which is a function of the reservoir temperature, said apparatus also having the following characteristic features, separately or in combination:

1. The compound enclosed in the reservoir is constituted by mono-ammoniated silver chloride in divided state;

2. The device comprises a screen with meshes sufficient for retaining in the reservoir the particles of the complex compound contained therein, while the gas is allowed to pass;
3. Between the reservoir and the exit canalization for the ammonia gas a capillary passage as such is arranged for upholding a constant ammonia rate for a determined dissociation pressure;
4. The reservoir is enclosed by an electric heater element as well as a thermo-sensitive resistor intended for regulating the energy supplied to the element for upholding the temperature at precisely the desired value;
5. The device comprises means allowing connection of the reservoir in a sealing manner either directly to a source of ammonia gas for recharging the compound contained therein or to an appliance using said gas, optionally through a regulating capillary passage as provided under item 3.

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REPRESENTED BY THE ADMINISTRATION NATIONALE DE L'AERONAUTIQUE ET DE
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Jh. Monnier

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